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3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

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Patents ADP number *(if you know it)*

If the applicant is a corporate body, give the country/state of its incorporation

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(A COMPANY REGISTERED IN ENGLAND)

4. Title of the invention

FLAME RETARDANT PRODUCT

5. Name of your agent *(if you have one)*

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

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Description 7

Claim(s) 2

Abstract 1

Drawing(s) 0

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Request for preliminary examination and search *(Patents Form 9/77)* 1

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Signature Alexander McPherson Date 13/7/02

12. Name and daytime telephone number of person to contact in the United Kingdom

ALEXANDER McPHERSON
01606 852537

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FLAME RETARDANT PRODUCTS

This invention relates to flame retardant products.

Flame retardant products exhibiting intumescent properties are well known. Such flame retardant products are incorporated in many functional host materials, especially thermoplastic polymer compositions. One such flame retardant product is a blend of a phosphoric acid producing catalyst, a charring agent and a blowing agent. The catalyst is a compound, e.g. ammonium polyphosphate, which when exposed to flame yields phosphoric acid. The charring agent can be a polyhydric alcohol, e.g. pentaerythritol, which decomposes and reacts with phosphoric acid to form a carbonaceous char. The blowing agent, e.g. melamine, when exposed to flame produces a non-flammable gas (e.g. N_2) which serves to foam and expand the carbonaceous char.

The above mentioned three component flame retardant products are powder additives which have processing limitations as they do not blend well with many functional host materials, e.g. thermoplastics. In order to overcome these processing problems there have been attempts to encapsulate the flame retardant additives in inert polymers. However, there is a disadvantageous limit on the amount of such encapsulated flame retardant product that can be incorporated in the host materials and the encapsulants themselves are generally flammable materials.

Proprietary flame retardant products have appeared on the market which typically are reaction products of pentaerythritol and phosphate esters. These flame retardant products are melt blendable with host materials such as thermoplastic polymers. However, such proprietary flame retardant products have to be used in combination with other flame retardants. Furthermore, such proprietary materials do not contain blowing agents and so do not have the advantages of char foaming and expanding.

An object of the present invention is to provide a unique flame retardant product which overcomes the problems of known flame retardant products in that it is more

rendily blendable with many functional host materials, particularly thermoplastic compositions, and thus imparts a higher degree of flame retardancy to the host materials.

According to the present invention, there is provided a flame retardant product comprising a phosphorous containing material which decomposes to produce phosphoric acid when exposed to flame and an oxygenated heterocyclic thermoplastic resin.

The phosphorous containing material preferably is selected from ammonium polyphosphate, sodium polyphosphate, potassium polyphosphate, melamine polyphosphate, melamine phosphate or mixtures thereof.

Advantageously, the phosphorous containing material is a mixture of ammonium polyphosphate and melamine phosphate.

The oxygenated heterocyclic thermoplastic resin preferably is an aldehyde resin.

Suitably, the thermoplastic resin is a condensation product of urea, formaldehyde and isobutyraldehyde.

The flame retardant product of the invention may contain a blowing agent, suitably melamine or urea.

Preferred composition ranges for the flame retardant product are 25 to 60% by weight oxygenated thermoplastic resin; 0 to 75% by weight ammonium polyphosphate; 0 to 75% by weight melamine phosphate and 0 to 45% melamine.

In the flame retardant product according to the invention, all other ingredients are encapsulated in the oxygenated heterocyclic thermoplastic resin.

In this specification an aldehyde resin can be a resin prepared according to the process described in US Patent 4720751 (BASF) wherein the resin is a condensation product of an urea and a CH-acidic aldehyde or a resin prepared according to the process

described in US Patent 4243797 (BASF) wherein the resin is a condensation product of urea, formaldehyde and a CH-acidic aldehyde. CH-acidic aldehydes are those where the carbon adjacent to the carbonyl group carries one or two hydrogen atoms.

From another aspect, the invention is a functional host material incorporating the flame retardant product as claimed herein. The host material may contain an amount of 5 to 90% by weight, preferably 10 to 45% by weight of the flame retardant product. Higher inclusions may be desirable for masterbatches and systems requiring intumescent functionality.

Suitable host materials include thermoplastic polymers, thermosetting polymers, reconstituted wood products and solvented systems (i.e. where the flame retardant product is dissolved in a solvent or mixtures of solvents).

Preferred host materials are polyolefins, particularly polypropylene.

Host materials containing the flame retardant product of the invention can be used in the manufacture of a wide variety of products and components for use in the electronic, construction and transport industries and can be incorporated into many structures including fire doors, vehicle passenger compartments, aircraft passenger and cargo areas as well as cargo storage containers and aircraft galley equipment, railway and underground carriages, cable trays (to prevent both loss of signal through the cable and passage of fire and heat along the cable tray itself), marine bulkheads, compressed gas and building structures.

Embodiments of the invention will now be described by way of example.

In the following examples of the invention the functional host material is polypropylene. The examples show that the Limiting Oxygen Index (LOI) of polypropylene incorporating the flame retardant product of the invention is increased. Since oxygen forms approximately 21% of normal atmosphere, thermoplastic polymers which have an LOI of 21% or less usually burn freely in air. If the inclusion of a material into the polymer increases the LOI of the polymer then this means that some degree of flame retardance is imparted to the polymer. As the LOI of the

polymer increases above 21% then the polymer becomes increasingly difficult to ignite and also increasingly likely to self extinguish. Generally speaking, once the LOI increases to above 30% then the polymer in effect is considered to be non-flammable and an LOI of 25% indicates good flame retardancy.

Successful polypropylene formulations containing a variety of examples of the flame retardant product of the invention are illustrated in the table below. All of the formulations contain the essential ingredients (a) the thermoplastic resin and (b) the phosphoric acid source (ammonium polyphosphate and/or melamine phosphate and some of the formulations also include melamine as a blowing agent).

The flame retardant product of the invention is not a simple combination of the powdered components but rather it is an extrudate. The ammonium polyphosphate, melamine phosphate and melamine are effectively encapsulated in the oxygenated heterocyclic thermoplastic resin during the extrusion process. This extrudate is normally produced as a chip (but with different equipment it could be made as a pellet or prill if required). The chip can be milled to a powder if this is considered desirable.

The finished flame retardant product has the appearance of a piece of dull white plastic. The product is virtually dust free and the chip size can be varied to suit end use requirements. The flame retardant product of the invention is a melt blendable product. The oxygenated heterocyclic thermoplastic resin casing is both part of the integral flame retardant mechanism but also makes the product melt blendable with many host materials. Compared to the traditional blends of flame retardants there is no pentaerythritol present. The oxygenated heterocyclic thermoplastic resin is the charring agent as well as giving the flame retardant product its melt blendable property.

It is to be noted that the flame retardant product of the invention is not a reaction product of its ingredients but rather is a physical blend of the ingredients. To our knowledge, no other non-halogen flame retardant uses this method of having an oxygenated heterocyclic thermoplastic resin incorporated which is part of the flame retardant system. Other flame retardant systems normally use inert polymers to either

encapsulate the products or as an inert backbone onto which the flame retardant molecule is grafted.

In Table 1 below, the test samples were produced by compression and in Table 2, the test samples were produced by injection.

In the Tables, the following terms have the following meanings:

- PP means polypropylene

- APP means ammonium polyphosphate

- MP means melamine phosphate

- the "Level" column indicates the % w/w inclusion of the flame retardant product in untreated polypropylene.

- MFI refers to the Melt Flow Index – this gives an indication of how difficult the flame retardant addition makes the resulting polymer composition to process (in general the lower the MFI, the more difficult the polymer composition is to process). The MFI conditions were 190°C and a weight of 2.16kg.

- UL94 refers to a standard test of the Underwriters Laboratory.

The ammonium polyphosphate used in the examples was Exolit AP422 from Clariant.

The oxygenated heterocyclic thermoplastic resin used was Laropal A81 which is an aldehyde resin obtained from BASF. The aldehyde resin, Laropal A101, again obtainable from BASF also could be used.

The melamine phosphate used was Melapur MP obtainable from DSM Melapur. The melamine phosphate provides both a phosphoric acid source for the char formation and a source of melamine and so provides dual function.

Table 1

Example	Aldehyde Resin	APP	MP	Melamine	Level	LOI	MFI
Blank PP	0	0	0	0	0	17	30
1	40	40	10	10	10	19	
2	40	0	60	0	20	19	
3	40	40	0	20	20	21	22.5
4	40	50	5	5	20	23	
5	40	40	20	0	20	23	22
6	40	40	10	10	20	25	20
7	35	55	5	5	20	27	17
8	40	30	15	15	30	21	
9	40	50	5	5	30	28	23.5
10	40	40	10	10	30	29	
11	35	55	5	5	30	31	23.5
12	35	45	10	10	20	23	
13	35	50	7.5	7.5	20	22	
14	35	65	0	0	20	22.5	
15	45	55	0	0	20	20	

Table 2

Example	Aldehyde Resin	APP	MP	Melamine	Level	LOI	UL94 (1.6mm)
16	40	40	10	10	30	24.8	Full Burn
17	35	55	5	5	30	31.3	Full Burn
18	35	45	10	10	30	31.3	V0
19	35	50	7.5	7.5	30	33.2	V0

The quantities expressed in the Tablea are weight percentages.

Observations

(i) It is possible to improve the LOI of polypropylene using an intumescent system comprising only two components (ketone resin + MP – Ex 2) and (ketone resin + APP – Examples 14 & 15).

(ii) It is also possible to improve the LOI of polypropylene with three component systems (Examples3 and 5).

(iii) The most successful results are with four component intumescent systems (Examples 7, 9, 10, 11, 12, 13, 16, 17, 18 and 19).

(iv) Table 2 shows that formulations can be prepared to achieve LOI results of 33.2.

(v) UL94 VO ratings can be achieved at a thickness of 1.6mm.

CLAIMS

1. A flame retardant product comprising a phosphorous containing material which decomposes to produce phosphoric acid when exposed to flame and an oxygenated heterocyclic thermoplastic resin.

2. A flame retardant product as claimed in claim 1 wherein the phosphorous containing material is selected from ammonium polyphosphate, sodium polyphosphate, potassium polyphosphate, melamine polyphosphate, melamine phosphate or mixtures thereof.

3. A flame retardant product as claimed in claim 2 wherein the phosphorous containing material is a mixture of ammonium polyphosphate and melamine phosphate.

4. A flame retardant product as claimed in any one of the preceding claims wherein the oxygenated heterocyclic thermoplastic resin is an aldehyde resin.

5. A flame retardant product as claimed in claim 4 wherein the thermoplastic resin is a condensation product of urea, formaldehyde and isobutyraldehyde.

6. A flame retardant product as claimed in any one of the preceding claims containing a blowing agent which produces a non-flammable gas when exposed to flame.

7. A flame retardant product is claimed in claim 6 wherein the blowing agent is melamine or urea.

8. A flame retardant product comprising 25 to 60% by weight oxygenated thermoplastic resin; 0 to 75% by weight ammonium polyphosphate; 0 to 75% by weight melamine phosphate and 0 to 45% melamine.

9. A flame retardant product according to any one of the preceding claims wherein all other ingredients are encapsulated in the oxygenated heterocyclic thermoplastic resin.

10. A flame retardant product substantially as hereinbelow described with reference to the Examples.

11. A functional host material incorporating a flame retardant product as claimed in any one of the preceding claims.

12. A functional host material as claimed in claim 11 wherein the flame retardant product is present in the host material in an amount of 5 to 90% by weight.

13. A functional host material as claimed in claim 12 wherein the flame retardent product is present in the host material in an amount of 10 to 45% by weight.

14. A functional host material as claimed in claim 13 wherein the host material is selected from thermoplastic polymers, thermosetting polymers, reconstituted wood products and solvents.

15. A functional host material as claimed in claim 14 wherein the host material is a polyolefin.

16. A functional host material as claimed in claim 15 wherein the polyolefin is polypropylene.

ABSTRACT

FLAME RETARDANT PRODUCTS

A flame retardant product comprises a phosphorous containing material which decomposes to produce phosphoric acid when exposed to flame (e.g. ammonium polyphosphate and/or melamine phosphate) and an oxygenated heteroxyclic thermoplastic resin (e.g. an aldehyde resin). A blowing agent (e.g. melamine) also may be included in the flame retardant product. The thermoplastic resin encapsulates the other ingredients thus making the flame retardant melt blendable with the host material in which it is incorporated (e.g. thermoplastic polymers, thermosetting polymers, solvented systems and reconstituted wood products).